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(19) (CA) **CANADIAN PATENT** (12)

(54) Use Of a Submersible Viscometer in The Primary
Separation Step Of the Hot Water Process For
Recovery Of Bitumen From Tar Sand

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1 "USE OF A SUBMERSIBLE VISCOMETER IN
2 THE PRIMARY SEPARATION STEP OF
3 THE HOT WATER PROCESS FOR
4 RECOVERY OF BITUMEN FROM TAR SAND"

5 ABSTRACT OF THE DISCLOSURE

6 The hot water process is controlled in response to
7 viscosity measurements taken in situ in the middlings in the primary
8 separation vessel. The viscosity in the middlings is found to vary.
9 Therefore, the layer of maximum viscosity is located and the viscosity
10 at this depth is monitored. Adjustments are made to the process to keep
11 this maximum viscosity below a pre-determined limit.

FIELD OF THE INVENTION

This invention relates to an improvement of the flotation-sedimentation step, for recovering bitumen from a tar sand slurry in a primary separation vessel, which step forms part of a conventional tar sand plant circuit. More particularly, it relates to the manner in which the viscosity of the middlings is measured and to the utilization of the measurements so obtained to guide adjustments to the process conditions.

BACKGROUND OF THE INVENTION

Tar sands, also referred to as oil sands and bituminous sands, contain a heavy oil usually referred to as bitumen.

There are tar sand deposits, in the Athabasca region of Alberta, which are today being commercially exploited. In connection with these operations, the tar sand is first mined and the bitumen is then extracted from the mined tar sand by a process called the hot water process. The extracted bitumen is subsequently upgraded by refinery-type processing, to produce synthetic crude.

The tar sand is a mixture of sand grains, connate water, fine minerals of the particle size of clay, and bitumen. It is commonly believed that the connate water envelopes the grains of sand, the fine solids are distributed in the water sheaths, and the bitumen is trapped in the interstitial spaces between the water-sheathed grains.

The hot water process is now well described in the patent and technical literature. A schematic of the circuit is shown in Figure 3.



In broad summary, this process comprises first conditioning the tar sand, to make it amenable to flotation-sedimentation separation of the bitumen from the solids. Conditioning involves feeding mined tar sand, hot water (180°F), an alkaline process aid (usually NaOH), and steam into a rotating horizontal drum, wherein the ingredients are agitated together. Typically, the amounts of reagents added are in the following proportions:

tar sand	- 3250 tons
hot water	- 610 tons
NaOH	- 4 tons (20% NaOH)

Enough steam is added to ensure an exit temperature of the mixture from the drum of about 180°F. The residence time in the drum is typically about 4 minutes.

During conditioning, the mined tar sand (in which the bitumen, connate water and solids are tightly bound together) is converted into an aqueous slurry of porridge-like consistency, wherein the components are in loose association.

The slurry leaving the drum is screened, to remove oversize material, and then flooded or diluted with additional hot water. The diluted slurry typically comprises 7% by weight bitumen, 43% water, and 50% solids. Its temperature is typically 160 - 180°F.

The diluted slurry then is transferred to the primary separation step, wherein it is temporarily retained in a large separation vessel having a cylindrical upper section and conical lower section. (This vessel is hereafter referred to as the "PSV" - for 'primary separation vessel'.) The vessel is similar to a thickener and has a rake system in its lower end, to assist in discharging the sand bed which accumulates there. The slurry is retained in the PSV for about 45 minutes in a quiescent condition.

During this interval, air bubbles, incorporated into the dilute slurry during conditioning, attach themselves to the bitumen, which is in the form of flecks or globules. Most of the aerated globules are buoyant and they rise through the slurry, to collect at the upper surface in the form of a froth. This froth is referred to as primary froth.

Most of the coarse solids, primarily being sand particles, sink through the slurry, are concentrated in the conical bottom end of the vessel, and are discharged through a bottom outlet. This stream is discarded as tailings (known as the 'primary tailings').

Not all of the bitumen becomes sufficiently aerated so as to rise and join the primary froth. Some of this non-buoyant bitumen is lost with the primary tailings. Most of it, together with a large part of the fines, collects in the mid-section of the PSV. This aqueous mixture is termed "middlings".

A dragstream of the middlings is withdrawn from the vessel and is fed into subaerated flotation cells, wherein secondary separation is practised. Here the middlings are subjected to vigorous agitation and aeration. Bitumen froth, termed "secondary froth", is produced.

Typically, the primary and secondary froths have the following compositions:

	<u>Primary (% by weight)</u>	<u>Secondary (% by weight)</u>
Bitumen	66.4	23.8
Solids	8.9	17.5
Water	24.7	58.7

1 It will be noted that the secondary froth is considerably
2 more contaminated with water and solids than the primary froth. One
3 seeks to minimize this contamination, as the froth stream requires
4 downstream treatment, to remove solids and water, before it can be fed
5 to the upgrading process.

6 It is therefore desirable to operate the process so that
7 as much of the bitumen as possible reports to the primary froth.

8 In summary then, the contents of the PSV may be described
9 as existing in the form of three sequential layers. At the base, one
10 has the tailings - this is primarily sand with some water and a minor
11 amount of bitumen entrained therein. Above this layer, one has the
12 middlings - this is water containing fines and insufficiently buoyant
13 bitumen. But passing downwardly through the middlings are many coarse
14 sand particles and rising through the layer are some buoyant bitumen
15 globules. And at the top, one has the froth.

16 Of particular interest are the well-aerated bitumen
17 globules, which should rise and form the primary froth, which is the
18 main commercial product of the process. These globules must make
19 their way up through the middlings.

20 If the middlings are too viscous, the well-aerated bitumen
21 globules may fail to achieve the needed upward velocity, and may end
22 up being discharged with the primary tailings or being withdrawn
23 with middlings for treatment in the secondary separation circuit. If
24 the globules exit with the primary tailings, they are lost entirely
25 from the process. If they are removed to secondary recovery, they
26 will be recovered in the form of poor quality froth.

1 At this point, it is appropriate to point out: (1) that
 2 the nature of the tar sand feed is variable; and (2) that the capability
 3 of the hot water process to extract the contained bitumen is significantly
 4 affected by the nature of the tar sand feed.

5 More particularly, the tar sand may contain a relatively
 6 high content of bitumen and a relatively low content of fines. This type
 7 of feed is referred to as "rich" tar sand. Alternatively, the tar sand
 8 may be relatively low in bitumen and high in fines. Such a feed is
 9 referred to as "lean" tar sand.

10 Typically, a "rich" tar sand can have a composition as
 11 follows:

12	14.44%	bitumen
13	0.36%	water
14	85.2%	total solids

15 Typically, a "lean" tar sand can have a composition as
 16 follows:

17	7.56%	bitumen
18	0.5%	water
19	91.84%	total solids.

20 The percentage fine solids (-44 μ solids in the total
 21 solids) can range from 5% for rich tar sands to as high as 25% for some
 22 lean tar sands.

23 In general, the rich tar sand feeds yield high primary
 24 froth recoveries. The lean feeds give low primary froth recoveries.
 25 Stated otherwise, the lean feeds are difficult to process with the hot
 26 water extraction procedure; they do not contain much bitumen and such
 27 bitumen as they do contain is difficult to extract.

1 This is partly because the lean feeds contain many fines,
2 which interfere with the flotation-sedimentation separation taking place
3 in the middlings layer of the PSV. In addition, the flecks or globules
4 of bitumen which appear in the PSV middlings, when lean tar sand is
5 the feed, are minute compared to the globules that are there when the
6 tar sand feed is rich. These minute flecks do not rise as readily as
7 the larger flecks.

8 If the fines content in the middlings becomes high, the
9 flotation mechanism can literally become inoperative. There is so little
10 primary froth being produced that the process performance is unacceptable.
11 In this instance, the contents of the PSV may have to be jettisoned and
12 the process started up again.

13 There are a number of courses of action open to the
14 operator by which he may adjust and alleviate undesirable process conditions
15 in the PSV arising from the nature of the tar sand feed. For example, he
16 can:

- 17 - adjust the rate of NaOH addition; or
- 18 - adjust the rate of water addition to the conditioning
19 or flooding steps; or
- 20 - blend some better quality tar sand in with the lean
21 tar sand, to provide a blended feed; or
- 22 - vary the residence time or temperature in the
23 conditioning drum.

24 A crucial matter, though, is to know when to make these
25 adjustments and to what extent the adjustment should be made. This
26 requires that a process parameter be monitored, which parameter gives
27 the operator a useful guide on which to base the adjustments.

1 It has heretofore been broadly taught in the prior art
2 that the viscosity of the middlings can be monitored and maintained
3 within staged ranges, to optimize the primary bitumen froth recovery
4 from the PSV. This teaching appears in Canadian patent 889,823, filed
5 by Graybill et al. Also of interest are Canadian patents, 889,825
6 and 841,581.

7 However, in accordance with conventional practise, the
8 viscosity has been monitored in one of the following ways:

- 9 - withdrawing a sample from the middlings dragstream
10 and measuring the sample viscosity with an appropriate
11 instrument; or
- 12 - lowering a sampler into the middlings, taking a grab
13 sample, and measuring the sample viscosity with an
14 appropriate instrument; or
- 15 - applying density measurements to either of the
16 foregoing samples and assuming that the viscosity
17 varies proportionately with the density.

18 Now, there are certain shortcomings associated with
19 these prior art practises.

20 If one samples the middlings dragstream, one must assume
21 that this sample - taken at one level of the PSV (there is usually
22 only a single outlet in the PSV wall) - is representative of the entire
23 column of PSV middlings.

24 When one attempts to measure the viscosity of this sample,
25 one is dealing with a mixture of sand, oil, clay, and water. The sand
26 and oil begin to settle and rise instantaneously. In addition, the
27 mixture is not static. It is impossible to duplicate the flow and
28 turbulence conditions which exist within the PSV.

1 Perhaps for these reasons, the industry has moved toward
2 measuring the density of the sample and assuming that the trend of
3 viscosity will follow the trend of density.

4 SUMMARY OF THE INVENTION

5 In the fundamental step of this invention, the viscosity
6 of the middlings is taken in situ in the PSV with a submersible viscometer.

7 In the testing which led up to this invention, when this
8 was done the following discoveries were made:

- 9 (1) that the viscosity varies strikingly at various
10 depths in the middlings in the PSV;
11 (2) that while the in situ-measured viscosity in the
12 PSV may vary significantly, the density of the
13 middlings when measured in connection with grab
14 samples may vary very little - therefore there does
15 not appear to be a useful correlation between the
16 two that may be relied on; and
17 (3) that the viscosity measurements obtained in situ
18 vary significantly from those obtained by taking
19 grab samples at the same depth in the PSV and
20 measuring the viscosity of the grab samples in a
21 conventional instrument external of the PSV.

1 Stated otherwise, it has been found that it is necessary to
2 measure the viscosity of the middlings in the dynamic environment of the
3 PSV contents, in order to obtain reliable and useful measurements. It is
4 postulated that the currents which arise in the PSV (from the continuous
5 entry of fresh slurry, the withdrawals of the tailings and middlings streams,
6 and the influences of dropping solids and rising bitumen), together with the
7 presence of the solids at the point of testing, combine to create a unique
8 and depth-variable viscosity regime in situ which differs in kind from that
9 which may be measured in grab samples and dragstreams. It is this unique
10 in situ viscosity regime which must be monitored in order to give the desired
11 guidance for process control.

12 In a preferred embodiment, one may "hunt" out the maximum
13 viscosity level in the middlings in the PSV by moving the submersible
14 viscometer vertically and taking measurements at different levels. One
15 then alleviates the undesirable process conditions by monitoring
16 viscosity at this level and making one or more process adjustments,
17 as previously described, to control said maximum viscosity and bring it
18 close to a pre-determined desired value.

19 Broadly stated, the invention is an improvement in the primary
20 separation step of the hot water process for extracting bitumen from tar sand
21 in a primary separation vessel, wherein the bitumen floats upwardly in a
22 tar sand slurry to form a froth layer, the coarse solids drop to form a
23 tailings layer, and a middlings layer is formed between the froth and the
24 tailings. The improvement comprises: providing a submerged viscometer in
25 the middlings layer and actuating said viscometer to measure the viscosity
26 of the middlings at one or more levels in the vertical column of middlings
27 and produce signals, external of the vessel, which are indicative of said
28 measurements; taking sufficient measurements to determine the viscosity of
29 the region of maximum viscosity within the middlings layer; and adjusting
30 the viscosity of the middlings in response to said signals to maintain the
31 maximum viscosity in the column below a predetermined value, whereby the

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- 1 flotation of the bitumen through the middlings layer to the froth layer is
- 2 substantially enhanced.

DESCRIPTION OF THE DRAWINGS

Figure 1 is a partially sectional side view of the viscometer used in connection with the invention;

Figure 2 is a sectional side view showing the viscometer suspended in the PSV of the pilot hot water process circuit used in developing the invention;

Figure 3 is a schematic showing the hot water process circuit;

Figure 4 is a plot of measured in-situ viscosity versus depth in the PSV at which the viscosity was measured, showing the variation in viscosity which is present in the PSV middlings at different levels, for a single tar sand feed treated in two ways - one without NaOH addition and the other with NaOH;

Figure 5 is a plot of measured density values for grab samples taken at different depths for the tar sand runs which generated the data for Figure 4; and

Figure 6a is a fanciful representation of the PSV contents during the run in which NaOH was not used;

Figure 6b is a fanciful representation of the PSV contents during the run in which NaOH was used.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The viscometer 1 used was of the oscillating torsional pendulum type. The particular viscometer used was obtained from Nametre Co., Edison, New Jersey and was identified as Model 7-006. This particular viscometer has a sphere 2 which vibrates at a certain frequency in air. When the viscometer is immersed in a viscous medium, there is a change or diminution in vibration amplitude, which is related to the drag on the sphere. The additional power required, to maintain the amplitude, with the sphere immersed, at its value in air, is a measure of the viscosity of the medium.

The mode of operation of this instrument is explained in an article entitled "New technique accurately measures low viscosity on-line" in Control Engineering, July, 1975, pp. 39 - 40, which article is incorporated herein by reference.

The viscometer 1 was enclosed in a waterproof housing 3. Protective threaded bars 4, adjustable in length, were screwed into the housing 3 and protruded downwardly beside the sphere 2, to protect it against contact with the wall 5 and rake 6 of the PSV 7. A tube 8 was attached to the housing 3, whereby the unit could be raised and lowered - conductive leads 12 extended through the tube 8 to the viscometer. The viscometer was adapted to produce a signal, indicative of the change in vibration amplitude exerted by the PSV fluid, which signal was a measure of the viscosity of the fluid in which the sphere 2 was vibrating.

The viscometer 1 is shown in Figure 2 as it was used in the PSV 7. This PSV was a small, non-commercial pilot unit. However, processing results in this pilot unit had previously been shown to correlate with processing results in applicant's full scale commercial PSVs.

The pilot PSV 7 was glass-sided, so that the action within could be observed.

The PSV 7 was part of a circuit illustrated in Figure 3. This circuit comprised a tumbler 9, in which tar sand was mixed with hot water, NaOH, and steam; and conditioned. The product slurry from the tumbler 9 was diluted with additional hot water in a pump box 10. The diluted slurry from the pump box 10 was transferred into the PSV 7 and retained there under quiescent conditions, to produce bitumen, froth, tailings, and middlings. Middlings were withdrawn from the PSV 7 and treated in a bank of sub-aerated flotation cells 11, to produce secondary froth and secondary tailings. The foregoing steps were conducted in accordance with conventional hot water process conditions.

Example 1

The pilot circuit was used to process a tar sand designated "A". This was known to be a poorly processing, lean feed. Two runs were made during which the feed was treated by the hot water process. One run was carried out with NaOH process aid having been incorporated in the slurry; the other run was carried out without NaOH. Viscosity measurements were made during each run using the viscometer 1 at different depths in the middlings in the PSV 7. Two curves or plots of measured in situ viscosity versus depth were developed. Plot 1 in Figure 4 involved the run without NaOH. Plot 2 in Figure 4 involved the run with NaOH. The details of the conditions and primary froth recovery results of the two runs are now set forth.

Tar Sand "A" composition:

9.8% bitumen

3.2% water

87.0% solids

21.3% fine solids (expressed as % of -44 μ solids
in the total solids)

Pilot Processing of Oil Sands "A"

Oil Sand Feed Rate - 630 g/s
 Slurry Temperature - 80°C
 Rate of Total Water Addition - 418 g/s

<u>NaOH Addition (wt. %)</u>	<u>Primary Bitumen Recovery (%)</u>
0.000	9.5
0.025	22.1

As shown by plot 1 for the run without NaOH, at a depth of about 0.4 m in the PSV, the viscosity measured with the viscometer was about 15 mPa.s. As the viscometer was lowered, the viscosity increased rapidly to 110 mPa.s. at a depth of 0.8 m, and then diminished to about 80 mPa.s. at a final depth of about 1.2 m.

Thus the PSV contents, when the PSV was operating on this lean tar sand A, were shown to be characterized by:

- a low viscosity at the upper end of the body of contents (as very little primary bitumen froth was generated by the poorly processing slurry in the absence of NaOH);
- changes in viscosity with depth;
- and a "plug" or layer of high viscosity middlings intermediate its ends.

The PSV contents were visually observed through the glass wall of the vessel. Figure 6a depicts what was observed. Again, there was only a thin layer of primary bitumen froth at the top end of the vessel contents and a viscous intermediate layer, which contained much bitumen.

1 The same tar sand A was then treated under the same
2 conditions as the Plot 1 run, except that in this second run a
3 conventional amount of NaOH was used. The in-situ viscosity versus
4 depth results are shown by Plot II in Figure 4. At the top of the
5 cell contents the viscometer 1 indicated a high viscosity (130 mPa.s.),
6 indicative of the thick bitumen froth layer which was produced. As the
7 viscometer was lowered to 0.3 m, it passed through the froth-middlings
8 interface and the measured viscosity dropped off sharply. The viscometer
9 1 indicated that the viscosity continued to decline to a limiting value
10 around 10 mPa.s. in the lower part of the vessel. There was no "plug"
11 of highly viscous middlings to hinder the rise of the bitumen globules.
12 An improved primary bitumen froth recovery was obtained in this run
13 as compared with the first run. Visual inspection during the run
14 indicated that the PSV contents were of the form shown in Figure 6b.
15 There was a thick froth layer and no noticeable viscous layer laden
16 with bitumen.

17 Thus there was correlation between the results indicated
18 by the in situ viscometer measurements and PSV performance as indicated
19 by the primary oil recoveries.

20 During the two runs, several grab samples were also taken
21 at depths corresponding with some of those at which the viscometer 1
22 took in situ measurements. Attempts to measure viscosity representative
23 of conditions within the PSV, on withdrawn samples, resulted in failure.
24 The above-noted problems, that is, the ascent of bitumen in the sample
25 jars, the rapid settling of coarse solids, and the impractical require-
26 ments for reproducing the flow and turbulence currents of the PSV,
27 caused such measurements to be abandoned.

1 In summary, these results show that:

2 (1) use of the submersible viscometer produces results
3 that indicate that there are viscosity changes that
4 occur within a PSV with depth;

5 (2) If high viscosity layers are developed in the PSV
6 middlings, they do trap bitumen and diminish primary
7 bitumen froth production; and

8 (3) These high viscosity layers can be eliminated by
9 adjusting process conditions, thereby improving
10 primary bitumen froth recovery.

11 In use, the signals emitted by the viscometer 1, submerged
12 in the middlings, are monitored and the viscosity of the middlings
13 are adjusted by altering one of the aforesaid process conditions, to
14 maintain the maximum viscosity in the middlings column below a pre-
15 determined value.

1 THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY
2 OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

3 1. In the primary separation step of the hot water process for
4 extracting bitumen from tar sand in a primary separation vessel, wherein the
5 bitumen floats upwardly in a tar sand slurry to form a froth layer, the coarse
6 solids drop to form a tailings layer, and a middlings layer is formed between
7 the froth and the tailings, the improvement comprising:

8 providing a submerged viscometer in the middlings layer and
9 actuating said viscometer to measure the viscosity of the middlings at one or
10 more levels in the vertical column of middlings and produce signals, external
11 of the vessel, which are indicative of said measurements;


12 taking sufficient measurements to determine the viscosity of the
13 region of maximum viscosity within the middlings layer;

14 and adjusting the viscosity of the middlings in response to said
15 signals to maintain the maximum viscosity in the column below a predetermined
16 value,

17 whereby the flotation of the bitumen through the middlings layer
18 to the froth layer is substantially enhanced.

19 2. The improvement as set forth in claim 1 comprising:

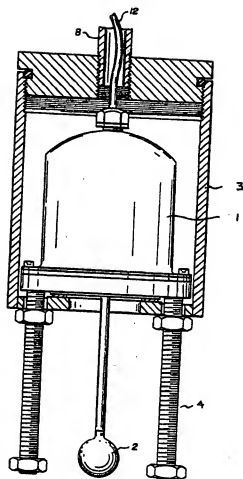
20 moving the viscometer vertically within the column of middlings
21 and locating and measuring the viscosity of the layer of middlings which
22 has the maximum viscosity.



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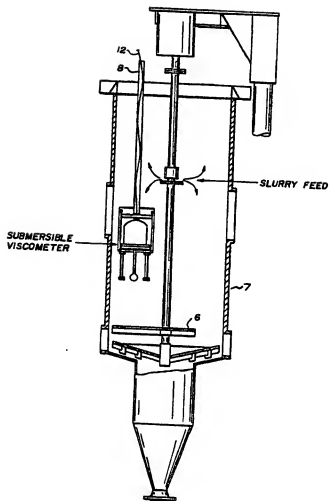
6-1

Fig. 1.

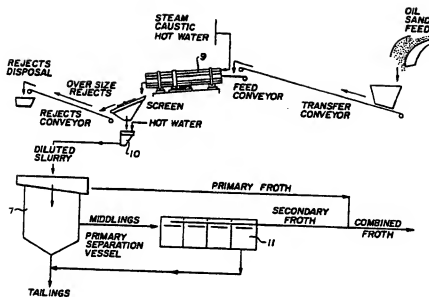


Patent agent:
E. P. Johnson

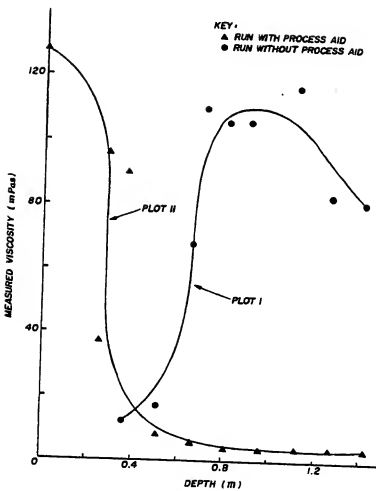
6-2

Fig. 2.

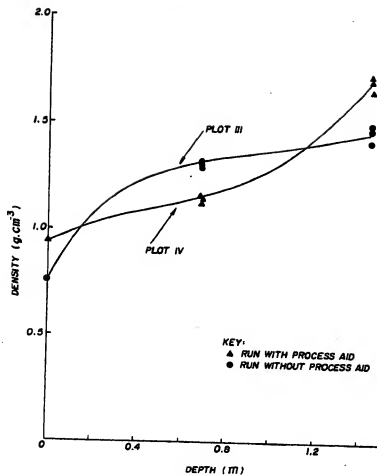
Patent agent:
C. P. Johnson

Fig. 3.

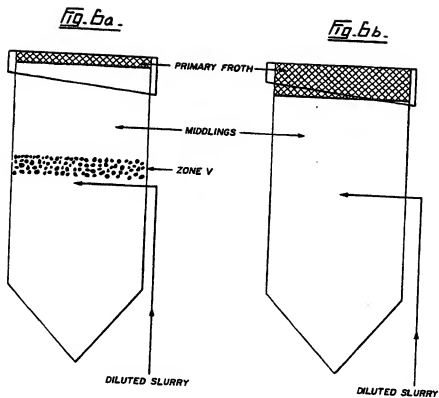
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Fig. 4.

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Fig. 5.

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